A133 Optimization of Headspace Solid Phase Microextraction Coupled With Gas Chromatography-Mass Spectrometry for Marijuana Profiling

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After attending this presentation, attendees will gain an understanding of Headspace Solid-Phase Microextraction (HS-SPME) methodology in order to profile trace samples of *Cannabis sativa L.*

This presentation will impact the forensic science community by providing preliminary results from a developed HS-SPME sampling method coupled with a Gas Chromatography/Mass Spectrometry (GC/MS) acquisition program for direct headspace sampling of cannabinoids and terpenes from solid samples of marijuana. This presentation will enhance the applicability of HS-SPME/GC/MS to controlled substance and trace evidence analysis.

Marijuana contains over 60 cannabinoids, of which the primary psychoactive component is Δ^9-Tetrahydrocannabinol (THC). Current analytical methods of detecting cannabinoids and other constituents of marijuana include solvent extractions coupled with gas chromatography and liquid chromatography. Limitations of solvent extractions may include the use of harsh chemical solvents, expense, sensitivity, and time-consuming extraction steps. A proposed solution that corrects for these limitations is a modified HS/SPME/GC/MS methodology to detect and profile the cannabinoid and terpenic constituents found in marijuana plant material.

In this research, three cannabinoid and seven terpene reference standards (cannabidiol, THC, cannabinol, α-pinene, eucalyptol, 3-methyl-3-cyclohexen-1-one, (R)-(+)-limonene, myrcene, β-cedrene, α-caryophyllene), commonly seen in marijuana, were selected for the optimization of HS-SPME extraction condition. Mixtures of the standards were first injected in liquid samples onto the GC/MS in order to optimize the acquisition method. Following GC-MS acquisition optimization, mixtures of the standards were then prepared for the optimization of HS/SPME extraction. Before HS/SPME, the mixture of ten reference standards was prepared in GC vials. The solvent was dried under a gentle nitrogen stream before being capped and closed. The samples were then extracted using a Polydimethylsiloxane (PDMS) 24-gauge, 100µm absorbent fiber, testing for the optimal extraction temperature and time range for maximum recovery of standards with minimal interference. Optimum extraction temperature was found to be 95°C while the optimum time of extraction was ten minutes to prevent highly volatile compounds from being degraded. When HS/SPME samples under high temperature (95°C) with longer extraction time was tested (e.g., 30 minutes extraction), four of the seven terpenes (α-pinene, myrcene, limonene, and eucalyptol) were unable to be extracted. Under the optimal HS/SPME/GC/MS condition, as little as 11.1ng solid cannabinoids in a GC vial were able to be extracted and detected. Regeneration of the PDMS fiber was performed by exposing the fiber in a GC inlet under 280°C for 30 minutes to eliminate carryovers.

This study indicates that the proposed HS/SPME/GC/MS method is highly sensitive, able to detect cannabinoids in nanograms. Commonly seen chemical constituents in marijuana samples in their solid form could be extracted and detected using the optimal condition of HS/SPME/GC/MS method. Future research will include sampling of actual marijuana plant material in order to further optimize the extraction and acquisition parameters of the methodology for use in drug chemistry and forensic science laboratories.

Marijuana, HS-SPME, Drug Analysis

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